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13. ABSTRACT (Maximum 200 words)

Brillouin light scattering has been successfully applied to investigate the elastic properties of thin films and membranes. Highlights of the project include the first evidence for the existence of "organ-pipe" type standing waves in supported films (silicon oxynitride and ZnSe) and free-standing membranes (SiN). These harmonics provide a direct means to investigate the longitudinal and transverse sound velocities and thereby to determine the C_{11} and C_{44} elastic constants. Evidence for excitations localized within a buried interface offers a previously unexplored approach to non-destructively characterize the elastic properties of sub-surface layers. The role of acoustic barriers offered by AlN in localizing elastic waves in GaN films and studies on amorphous ZrB_3 films reveal its transformation to hard nano-crystallites of the boride layer with dramatic enhancement of the elastic properties upon high temperature anneal. Analysis of the results was based on calculating the associated elasto-dynamic Green's tensor enabling the local density of states as well as the acoustic mode dispersion(s) to be determined. The observation of a range of acoustic excitations including dilatational and flexural modes in freestanding membranes, open new opportunities in the non-destructive study of high frequency acoustics of laminar structures and coatings.

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Final Report
DAAD 19-00-1-0396

Statement of Problem:

Thin films, coatings and nano-laminates are finding diverse applications ranging from protective coatings, improved wear at ambient and high temperatures and corrosion resistance. Concurrent with remarkable advances in the synthesis and fabrication of these laminar systems, there is a critical demand for determining their mechanical properties, assessing degradation and developing non-destructive techniques for use over their lifetime.

Determining the elastic properties of thin films and coating is challenging. Ultrasonic echoes are ineffective since the associated wavelengths are larger than typical film thickness'. Techniques such as vibrating reed, torsional and bulge testing require detachment of the film from the substrate while nano-indentation suffers from the need that the indentation depth be less than 10% of the film thickness. The latter is also sensitive to the substrate especially for hard films where the yield of the substrate upon indenting the over-layer affects the measurement.

In this project we have further developed Brillouin light scattering (BLS) as an effective non-destructive technique to probe the elastic properties of thin supported films that included insulating materials, wide band gap semiconductors and chemically ordered magnetic Co_3Pt layers. In addition, experiments related to the acoustic properties of freestanding nano-membranes were completed.

<u>Background:</u>

The ability to detect, surface and near surface localized acoustic waves by Brillouin light scattering provided the basis for this work. The technique offers high spatial resolution, and we developed this spectroscopy method to be readily applied in the investigation of coatings, supported layers, free-standing membranes and buried interfaces.

Brillouin light scattering (BLS) from thermally excited, high frequency acoustic excitations is emerging to be of great value in the study of acoustic and elastic properties of bulk and laminar structures[1-3]. For an opaque or semi-transparent medium, surface excitations polarized in the sagittal plane are the primary modes observed in these experiments. The corresponding scattering cross-sections are controlled mainly through surface corrugations generated by the modes (ripple mechanism) and only the wave-vector component parallel to the surface ($K_{//}$) need be conserved during scattering. On the other hand, for sufficiently thick transparent materials the volume elasto-optic (E-O) coupling mechanism is dominant and all components of momentum are conserved. For relatively thin films where the E-O mechanism is operative this condition on momentum is relaxed and results in the broadening of bulk phonons[4]. BLS has been successful in probing a variety of near surface excitations such as Rayleigh, Sezawa, Love and Lamb modes as well as guided acoustic resonances in thin films[5-14].

Summary of Important Results:

"Organ-pipe" harmonics in thin films
(Phys. Rev. B64, R081402 (2001) Rapid Communications)

Despite an extensive body of BLS experiments, standing acoustic wave excitations in laminar structures that are characterized only by wave-vector components perpendicular to the thin film surfaces ($K_\perp$) have received little attention. In this case due to the limited film thickness (d) and applicable acoustic boundary conditions, the allowed values of $K_\perp$ are quantized to a series of discrete values. Such constraints on $K_\perp$ are in contrast to modes with finite $K_{//}$ components that take on a continuum of values and describe traveling wave excitations that are evident in most BLS studies. Acoustic excitations that are characterized by only $K_\perp$ components, offer novel and previously unexplored opportunities to probe the elastic properties of thin film structures.

In this project we discovered and reported on the observation of a series of standing wave acoustic excitations akin to those of an organ pipe over an extended frequency range for scattering angles of 0° ($K_{//} = 0$). This special scattering arrangement was undertaken to measure the photons back-scattered essentially along the silicon oxynitride film normal and thus probe only $K_\perp$ components of acoustic excitations. An extensive number of discrete excitations mediated by the ripple scattering mechanism were observed well below the bulk LA mode which also separate into a distinct set of modes in the vicinity of the LA mode where the elasto-optic mechanism is more effective. Our results showed that the modes closely follow the behavior of the harmonics of an organ-pipe with a maximum displacement at the surface and a node at the film-substrate interface. This study represented the first observation of such "organ-pipe" excitations by Brillouin light scattering and is complementary to inelastic helium-atom scattering which probe similar, albeit short wave, excitations for very thin (2-20 monolayer) films[15]. We also showed that when the scattering angle is changed away from normal incidence, finite $K_{//}$ effects are operative and the modes become dispersive. The results were analyzed on the basis of a Greens function formalism where calculations for $K_{//} = 0$ show all of the important observed characteristic BLS features including the multitude of modes, their line widths and variations with film thickness and scattering angle. Moreover, the periodic absence of particular Brillouin peaks from these films was traced to the destructive interference between ripple-mediated scattering amplitudes at the film boundaries. The study provided a valuable step in classifying guided excitations of layered systems that also opened up a direct means for investigating the longitudinal elastic properties in structures, especially when the films are very thin.

High frequency standing wave harmonics in ZnSe films
(Phys. Rev B67, 075407 (2003)).

Underlying the experiments discussed above on silicon oxynitride was the surface ripple caused by the "organ-pipe" waves which provided the dominant light scattering channel out to 50 GHz. In this effort we selected ZnSe where the elasto-optic (E-O) mechanism dominates the scattering cross-section. This led to significant changes to the light scattering profile and provided previously unexplored results detailing important aspects of the scattering intensities. They included a complete analysis of the Brillouin scattering intensities that concomitantly considered contributions from elasto-optic (E-O) and surface/interface ripple channels in the film and substrate. These simulations, the first for standing wave modes in a thin film, provided direct insight into unusual aspects of the spectra including replicating the conspicuous presence of standing wave harmonics only beyond ~45 GHz in the spectra from ZnSe films and their weakness below 40 GHz. The relative importance of the different light scattering channels was highlighted and accounted for the absence of alternate Brillouin peaks as expected from a simple organ-pipe model.

Probing confined interfacial excitations in buried layers and interfaces
(Applied Physics Letters 80, 4501 (2202).

Characterization of the interface separating two layers remains a challenging problem in the field of thin film/ multi-layer growth and in applications relying on high quality microstructures. While the interface in such structures is important in determining the quality of the films, the transition from one material layer to the other is, in most cases, not abrupt. The properties of such transition layers are, in turn, important for they often act as a seed for the subsequent growth and therefore play a significant role in defining the properties associated with the ensuing film. The most common technique utilized in probing this buried region is transmission or scanning electron microscopy which, despite its high resolution, is burdened by being a destructive method. Amongst non-destructive techniques, inelastic laser light scattering has had very limited success in probing the interfacial region. This method relies on the detection of Stoneley acoustic waves[16] that are guided along the abrupt interface of two semi-infinite solid media in contact along a plane. The biggest drawback of exploiting Stoneley waves to probe the interfacial properties is that they exist only under strict restrictive conditions[16,17] wherein the shear acoustic velocities of the two adjacent layers are comparable. Thus the technique has been effective only in very few layered material combinations; for example in molybdenum films on fused quartz.[18]

In this experimental study we provided theoretical insight into our observation of an acoustic excitation localized by the interfacial region in the case where the two layers are separated by a transition layer. Brillouin light scattering (BLS) revealed this mode as a well defined low frequency excitation while simulations showed that its displacement amplitude falls off exponentially to zero in both the film and substrate with the excitation being guided by the transition layer. Thus while the mode is indeed localized by the

interface, it is not encumbered by stringent conditions on the shear velocities of the two layers like the Stoneley excitation[16]. Therefore observation and investigation of this excitation provided a previously unexplored avenue to non-destructively characterize the properties of buried interfacial layers of hetero-structures.

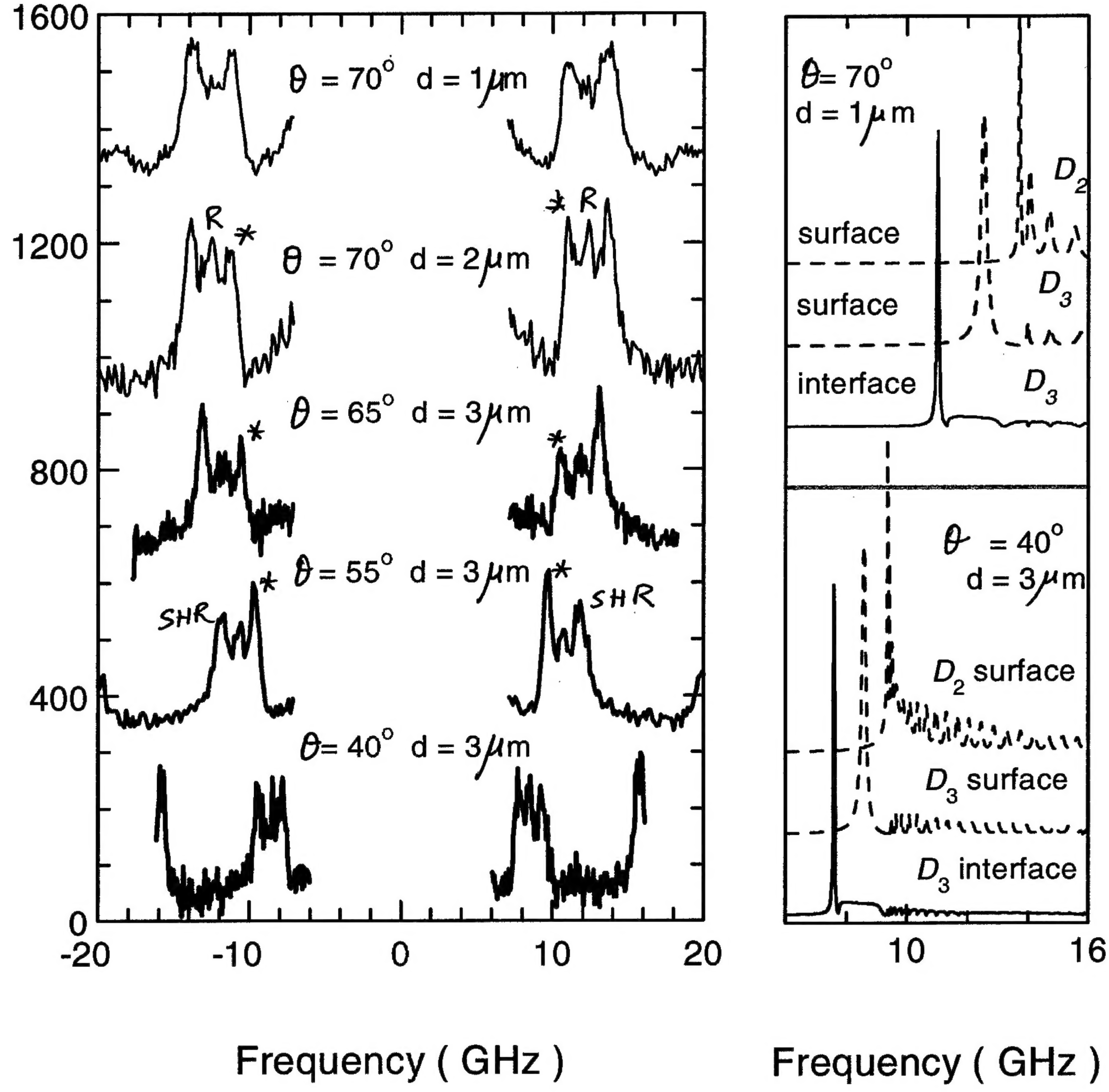

Figure 1: (a) Brillouin spectra for silicon oxynitride films on GaAs. The peak at the lowest frequency is identified (*) as a buried interfacial mode. The intermediate and high frequency modes are respectively the Rayleigh (R) and shear horizontal resonance (SHR). (b) Calculated density of states at the free surface (D_2 and D_3) and interface (D_3) for 1μm thick film at incident angle of 70 ° and for 3μm film at 40° with a transition layer thickness of 40nm.

Confinement and Transverse Standing Acoustic Resonances In Free-standing nano-Membranes
(Phys. Rev. B68, 115430 (2003)).

Practical micro-electromechanical system (MEMS) devices such as resonators[19], force detectors[20] and magnetometers[21] rely on the mechanical and elastic properties of free-standing membranes. In these applications, detection of changes in elastic variables such as the strain, deflection or torsion underlies the performance of the device. The independent elastic constants and moduli of the unsupported membrane hence become vital parameters in accurate interpretation of the measurements[22]. Conventional techniques to quantify these elastic constants are challenging since they demand careful

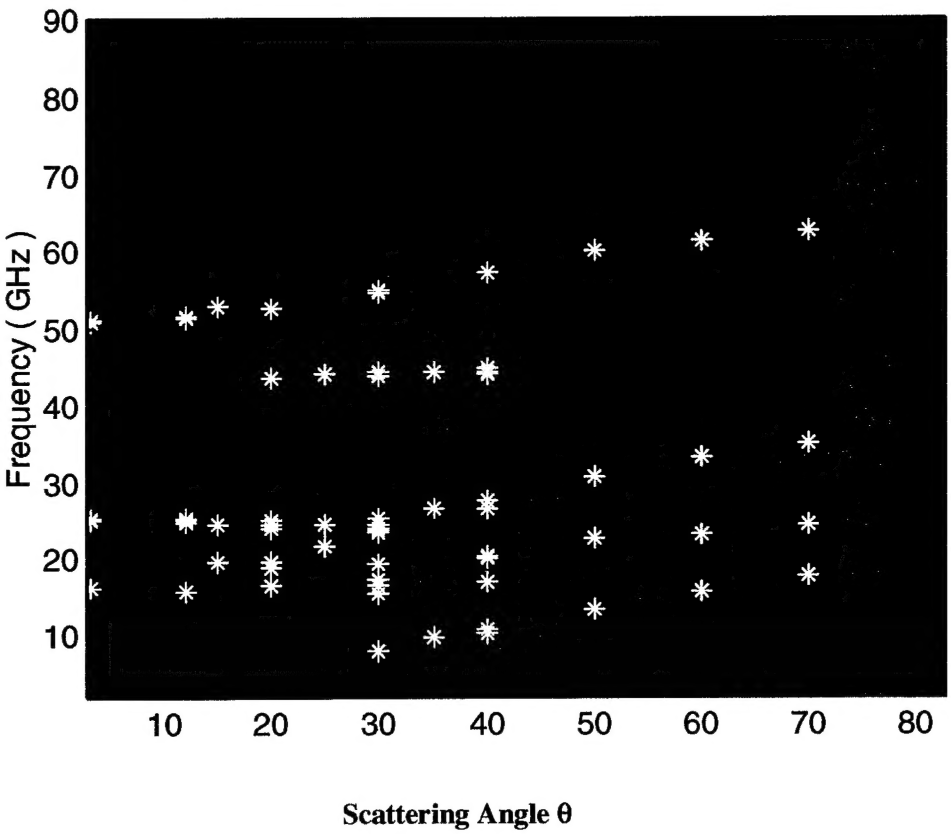

Figure 2: Dispersion curves for standing wave excitations in a 200nm thick *free standing* SiN film as a function of scattering angle θ, or equivalently the inplane wavevector $K_{//}$. The colors reflect the scattering intensities, with the darker (purple) shades representing stronger BLS signals. The * symbols indicate measured data.

loading of the MEMS device as well as specific knowledge about the dimensions and support conditions of the films. Detection of macroscopic mechanical resonances that may shed insight into these elastic properties on the other hand typically requires relatively large unsupported films thicker than ~1.5μm. There remain few reliable non-

destructive methods to determine the mechanical properties of *free-standing* nanometer-sized thin films.

In this study we investigated the application of Brillouin light scattering (BLS) to non-destructively determine the independent elastic constants of free-standing 100nm and 200nm thick SiN membranes. The study represents investigations of high frequency acoustic standing waves characterized by only discrete wave vectors ($K_\perp$) lying perpendicular to the membrane surfaces with negligible in-plane wave vector ($K_{//}$) components. The constraints on $K_\perp$ arise from the limited film thickness and are in contrast to modes with finite $K_{//}$ components that take on a continuum of values. While the series of $K_{//} \sim 0$ standing wave modes divide into pure longitudinal or transverse polarized excitations, transformation of these $K_\perp$ excitations to modes with finite in-plane wave vectors reveal strong dispersion of the N=2 order *transverse* resonance leading to strong interaction with resonances of longitudinal symmetry. At finite scattering angles, dilatational and flexural membrane modes were also evident. The mode dispersions and Brillouin intensities were well accounted for by the projected local density of phonon states and provide for the principal elastic constants of the nano-membranes. In addition it was shown all of the observed discrete acoustic resonances become strongly evanescent when the membrane was supported on a Si substrate leading to the absence of features in the light scattering spectra.

"Magneto-elastic effects in chemically ordered Co_3Pt"
(Journal of Applied Physics 91 (5) 2737 (2002))

The recently synthesized, chemically ordered, binary alloy Co_3Pt is an interesting system because of known correlations between its magnetic behavior and atomic order.[23-25] In particular with increasing growth temperature (T_g) the Co_3Pt layers transform from a compositionally-disordered, mixed fcc/hexagonal phase (T_g = 400K) to a compositionally-ordered, purely hexagonal phase (600K < T_g < 700K) and finally to a compositionally disordered fcc phase (T_g = 950K).[26] These modifications to the lattice structure are accompanied by corresponding changes in the magneto-optical Kerr signal, uniaxial magnetic anisotropy (K_U) as well as widths of the magnetic domains that all evolve as T_g was varied.[26-29] Motivated by recent findings that magneto-elastic interactions due to tetragonal strain contribute to the magnetic anisotropy energy in thin magnetic films,[30] we investigated the long wave acoustic properties of Co_3Pt films to determine the *elastic* properties that accompany the T_g-induced changes in crystalline structure. Knowledge of the elastic constants is important for they provide insight into the stability of these phases and enable relevant macroscopic strains in the layers to be evaluated. We found, as evidenced by the independent elastic constants C_{ij} determined from non-destructive Brillouin light scattering associated with near-surface and higher order acoustic waves, no significant variation in the elastic properties of Co_3Pt films grown at temperatures between 450 and 950K. These results thus suggest that macroscopic anisotropic strain is not responsible for the variations in the different magnetic properties reported from these films.

Enhanced elastic constants of nano-crystalline Zr-Si-B films

(Journal of Applied Physics 89 4349 (2001)).

Binary compounds in the Zr-Si-B system have been widely studied for structural and microelectronic applications. The high melting point, excellent corrosion resistance and immunity to oxidation at elevated temperatures characterize these materials as excellent hard materials[31-34]. In this work we discovered dramatic enhancements in the C_{11} and C_{44} elastic constants to accompany the conversion of ZrB_3 films deposited on Si to the nano-crystalline phase $Zr_{0.9}Si_{0.3}B_3$ when the films are annealed at high temperatures. These layers are also unusual in that the transverse sound velocity of the film and substrate are in near resonance which, in turn, renders the surface Rayleigh wave as the only acoustic excitation localized to the film; all higher order modes are evanescent. Despite the availability of strong decay channels, the Brillouin spectra reveal modes with very high velocities that are as high as five times the threshold sound velocity of the Si substrate.

These experiments were carried out in collaboration with Professors John Kovetakis and Ig Tsong at Arizona State University where the films were produced.

Acoustic barriers and observation of guided elastic waves in GaN-AlN structures by Brillouin scattering

(Phys. Rev. B63, 205302 (2001)).

GaN is a promising material for short-wavelength electro-luminescent devices.[35-37] The growth of GaN on AlN provides an avenue for the epitaxial growth of the dominant hexagonal GaN polytype onto Si substrates - a highly desirable feature for merging the advantages of the nitride material to the Si-based electronic industry.[38] In this study we reported on the acoustic properties of GaN/AlN heterostructures and showed that the AlN layer provides an active, high frequency, acoustic barrier that leads to effective localization of specific guided acoustic excitations to the GaN layer.

Observation of such surface and near-surface guided excitations by Brillouin light scattering enabled several of the principal elastic constants of the supported GaN layer to be determined non-destructively. One consequence of the presence of acoustic barriers in the GaN-AlN hetero-layers is the presence of the longitudinal guided resonance (LGR) propagating parallel to the film surface and a shear horizontal resonance (SHR) polarized in the plane of the surface. Calculations of the mode-density and -displacements disclose characteristics of the LGR and SHR and account for their localization and polarization features.

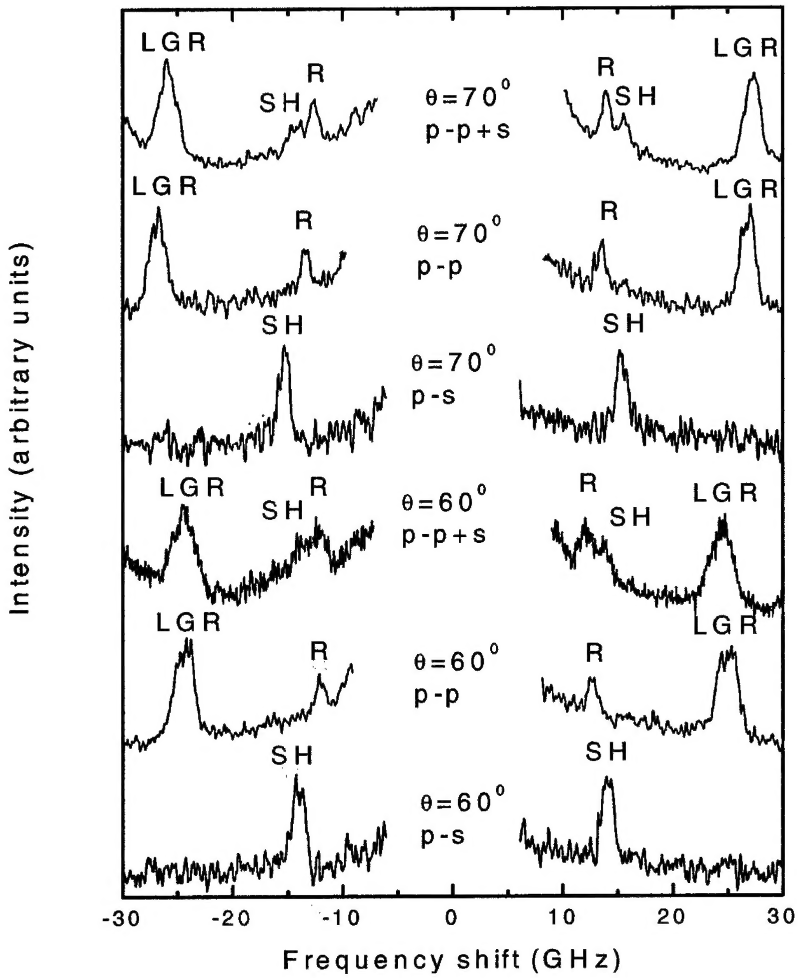

Figure 3: BLS spectra in backscattering from GaN/AlN/SiC/Si for p-p, p-s and p-p+s polarizations. LGR, SH and R identify the longitudinal guided resonance, the shear horizontal and Rayleigh excitations. The single crystal hcp-GaN layer of thickness 1.3μm was grown at Purdue University in the group of Professor Melloch on a sequence of designed buffer layers of 3C-SiC (200 nm) and 2H-AlN (200 nm) on Si(111) substrate.

Longitudinal guided modes (resonances) GaN/AlN structures. Figure 3 shows typical polarized (p-p), depolarized (p-s) and unpolarized (p-p+s) Brillouin spectra below 30 GHz from the GaN(1300 nm) /AlN (200 nm) /SiC (200 nm) /Si (111) stack for angle of incidences $\theta = 60°$ and $70°$. In addition to the principal Rayleigh mode (R) another mode, identified as the longitudinal guided resonance (LGR) of GaN, is present in the polarized (p-p) spectrum. The low frequency p-s depolarized spectra reveal the presence of the mode labeled SH that corresponds to the in-plane polarized acoustic wave.

The character of the LGR and SH modes were determined from the local density of phonon modes $n_i(\omega^2,q, z)$ evaluated within a Green's function formalism.[5,9] Here i (=1 – 3) identifies the mode polarizations where i = 3,2 are, respectively the sagittally polarized transverse and shear horizontal modes and i =1 the longitudinally polarized mode, $\omega(=2\pi\nu)$ the angular mode frequency and z the distance from the film surface where the mode density is calculated. In analyzing the response function of the sample we followed our previous work on DLC discussed above.

The GaN-AlN structures were produced by Professor Mike Melloch and his group at Purdue University.

Summary

Brillouin light scattering has been successfully applied to study the elastic properties of films, coatings and freestanding membranes. We have presented the first light scattering evidence for the existence of organ-pipe excitations. These excitations were also revealed by theoretical simulations based on calculating the associated Green's function tensor that allows the local density of states as well as their spatial mode displacements to be determined. The low frequency spectra were shown to be dominated by ripple mediated scattering. The study provided a valuable step in classifying guided excitations of layered systems that also opens up a direct means for investigating the longitudinal elastic properties of structures, especially when the films are very thin. Extensions to supported ZnSe films that support both surface ripple and elasto-optic scattering channels in the film and substrate showed that interference effects in the scattering amplitudes lead to the observed asymmetry in the BLS spectra below and above the film LA frequency. The calculations also accounted for suppression of Brillouin intensities of alternate standing wave harmonics.

We have also reported on standing longitudinal and transverse acoustic resonances (organ-pipe type modes) in free-standing SiN membranes. In addition the dilatational and flexural membrane modes were also observed. The mode displacements and Brillouin intensities were well accounted for by the projected local density of phonon states results and provided for the principal elastic constants of the nano-membranes.

BLS experiments have allowed an interface localized mode to be successfully observed. The mode - localized close to the interface - does not require the normal strict conditions of an abrupt interface for its existence. Its relative ease of observation via light scattering experiments provides a non-destructive approach to characterize the elastic properties of buried films and transition layers.

The elastic properties of a series of 100 nm thick Co_3Pt epitaxial films grown between 450 and 900 K were studied. In this range the structures ranged from compositionally disordered to chemically ordered hcp to a disordered fcc phase. The absence of a strong dependence of the independent elastic constants on the growth temperature was consistent with macroscopic anisotropic strain to be an unlikely source

for the large enhancements in the uniaxial magnetic anisotropies observed from films grown within a narrow temperature window between 600K and 700K.

BLS also revealed the presence of longitudinal and shear horizontal guided resonances in 2H-GaN films grown on AlN films deposited on Si. Theoretical simulations reveal the properties of these resonances and emphasize the important role of the AlN layer and Si substrate that act as acoustic barriers to these high frequency modes allowing for their localization inside the GaN layer. In addition, bulk LA and TA phonons as well as the surface Rayleigh excitation are observed. Observation of several distinct excitations in a single back-scattering experiment has also allowed for four of the five independent elastic stiffness constants (C_{11}, C_{44}, C_{66} and C_{33}) of the GaN layer to be determined. Investigations on the elastic properties of hard boride films and the transformations to their structural and mechanical properties that occur upon high temperature anneal were also conducted. Large enhancements in the principal elastic constants of the films when the as-grown ZrB_3 layers transform to nano-crystalline $Zr_{0.9}Si_{0.3}B_{0.3}$ when the films are annealed at 960°C were observed.

List of Publications and Technical Reports:

1. **Acoustic Barriers and Observation of Guided Elastic Waves in GaN-AlN structures by Brillouin scattering**, M. Chirita, R. Sooryakumar, R. Venugopal, J. Wan, M.R. Melloch, Physical Review B **63**, 205302 (2001).

2. **Elastic properties of nano-crystalline zirconium-silicon-boron thin films**, M. Chirita, H. Xia, R. Sooryakumar, J.B. Tolle et.al. Journal of Applied Physics **89**, 4349 (2001).

2. **Observation of organ-pipe acoustic excitations in supported thin films**, X. Zhang, R. Sooryakumar, A.G. Every and M.H. Manghnani, Physical Review B (Rapid Communications) **64**, R081402 (2001)

3. **Elastic properties of chemically ordered Co_3Pt thin films**, R. Bandhu, R. Sooryakumar, R.F.C. Farrow, D. Weller, Journal of Applied Physics **91** (5), 2737 (2002).

4. **Probing confined interfacial excitations in buried layers by Brillouin light scattering,** X. Zhang, R. Sooryakumar, Applied Physics Letters **80**, 4501 (2002).

5. **High frequency standing longitudinal acoustic resonances in supported thin films,** X. Zhang, R. Sooryakumar and B. Jonker Phys. Rev.B **67**, 075407 (2003).

6. **Confinement and transverse standing acoustic resonances in free-standing membranes,** X. Zhang, R. Sooryakumar and K. Bussmann, Phys. Rev. B **68**, 115430 (2003).

Scientific Personnel:

During the reporting period one student (Mr. Rudra Bhandu) was supported as a graduate research assistant. He will be completing all requirements for the Ph.D degree from Ohio State University within the next six months. In addition, Dr. Xinya Zhang a post-doctoral associate was supported briefly on this project. Professor R. Sooryakumar (PI) was supported with one month summer salary during the course of this project.

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